



Analysis of Burning Rate Phenomena and Extinguished Solid Propellants From an Interrupted Closed Bomb With Plasma Igniter

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Abstract

Interrupted closed-bomb analyses of M30 and JA2 propellants have been performed with the goal of understanding the phenomena responsible for the apparent augmentation of closed-bomb burning rates when plasma, rather than black powder, ignition is used. Burning rates of the propellants during plasma injection and after the plasma (to blowout pressures of 100 Mpa) have been estimated from the grain regression measurements and the P-t traces. Characterization of the extinguished grains has been performed in order to understand the interaction of the plasma with the propellant, including scanning electron microscopy (SEM) and Fourier-transform infrared (FTIR) analyses.

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1. Introduction

A 129-cm³ closed bomb capable of either plasma or conventional black powder ignition was previously used [1] to study the effect of electrothermal chemical (ETC) ignition on solid gun propellants. For M30, it was reported that a 33% increase in burning rate over the range of 100–220 MPa occurred with plasma ignition, relative to conventional ignition; the closed-bomb burning rate augmentation of JA2 was insignificant. Researchers in the international community have also reported plasma augmentation of the burning rate with nitramine-based composite propellant [2]. A goal of this work is to understand the phenomena responsible for the apparent increase in burning rate measured in the closed bomb when plasma, rather than black powder, ignition is used. The approach was to repeat key experiments from [1] using granular, 7-perf M30 and JA2, but interrupt the burning at 35, 75, and 100 MPa, and determine if there is an increase in the intrinsic burning rate of the propellant, or if other phenomena (e.g., fracture [3], porous burn, etc.) are involved.

The interrupted closed-bomb experiment offers a means of studying basic plasma-propellant interactions under well-defined conditions of plasma energy and power input, pressure profile, and proximity of the sample to the plasma. A second goal of this work is to characterize extinguished grains from both plasma- and conventionally ignited samples, in order to begin to understand the basic mechanisms of plasma-propellant interaction. Scanning electron microscopy (SEM) was performed to assess fracture, erosive burning, gas evolution, and melt layer thickness, and Fourier-transform infrared (FTIR) spectroscopy was used to detect and identify decomposition products and component depletion.

2. Experimental

2.1 Closed-Bomb Firings

Samples for closed-bomb analysis consisted of 7-perforated grains of M30 and JA2 with a diameter, length, and perforation diameter of approximately 0.75, 1.5, and 0.07 cm, respectively. Closed-bomb analyses of M30 and JA2 were performed in a 3.81-cm inside diameter (ID) closed bomb of 129 cm³, with a typical propellant charge weight of 32 g, with both conventional and plasma ignition. Details of the bomb configuration can be found in [1] and are only briefly described here. (In addition to the polyethylene capillaries used in [1], polyethylene terephthalate (mylar) capillaries have been used in this study.) For extinguished propellant measurements, the addition of the interface of the bomb

to the evacuation chamber yields a total closed-bomb volume of 150 cm³ (Figure 1). The expansion chamber consists of a 240-L tank with a 2.5-cm-diameter blowout area interfaced to the closed bomb.

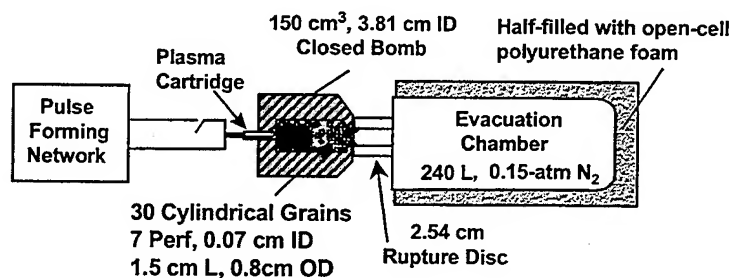


Figure 1. Experimental apparatus for extinguished closed-bomb experiments.

Rapid extinguishment of the propellants occurs due to the sudden expansion into the evacuated chamber. Soft capture of the propellant is achieved with a lining of thermally resistant polyurethane foam. In the case of plasma ignition, the plasma is channeled through a straw that is perforated to resemble a piccolo tube with 24 holes to enable uniform distribution of the plasma around the propellant. The plasma is generated by an electrical pulse to a nickel fuse wire that rapidly vaporizes. This results in ionization of the polyethylene liner, and a high-current discharge is sustained. The electrical pulse is generated with a 400-kJ capacitor-based pulse-forming network, with a charging voltage of 4 kV and output energy of up to 29.3 kJ. Acceptable firings yielded a power curve with a smooth rise and 1.2-ms duration; the peak power was typically 25–29 MW, and the plasma energy varied between 24–29 kJ. In the conventional mode, an electric match is used to ignite 0.6 g of black powder confined in a plastic straw with the propellant distributed concentrically in two tiers around the straw.

2.2 FTIR Spectroscopy

Microreflectance FTIR was used to obtain spectra of the propellant surface (i.e., about top 10 μ) with no modification of the sample. FTIR analyses were performed using a Mattson Polaris spectrometer operating at a resolution of 4 cm⁻¹. The reflectance spectra were obtained using a Spectra-Tech* microreflectance attachment with 32-μ IR objective and a signal averaging over 200 scans. Aluminum foil was used to collect the background spectra.

2.3 SEM

Micrographs of virgin and extinguished grains were examined to establish any morphological differences between grains ignited by conventional and plasma

* Thermo Spectra-Tech, 2 Research Dr., Shelton, CT 06484-0869.

sources. The grains were selected and cold fractured along the longitudinal grain axis to expose the burning surface of the perforations and a cross section of the burning surface and the propellant below this surface. The prepared specimens were sputter coated with gold-palladium. The lateral exterior burn surface was also examined for any differences.

3. Results

3.1 Closed-Bomb Firings

The P-t curves for the M30 and JA2 samples ignited with the plasma are shown in Figure 2, for blowout pressures of nominally 35 and 100 MPa. P-t curves for conventionally ignited M30 are also shown, and are evident from the longer time-to-burst of about 10 ms at 60 MPa. Also apparent is the uniform curvature due to the regular mass generation of the conventionally ignited propellant. In contrast, propellants ignited by plasma show a sharp increase in pressure within the first 1-2 ms due to the sudden plasma impulse, followed by a lower rate of pressure increase when normal propellant burning takes over.

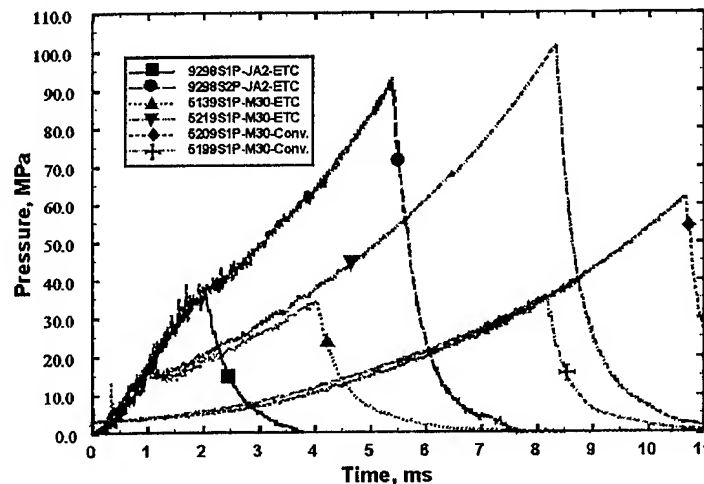


Figure 2. P-t curves for trials used in assessing post-plasma burning rate.

Originally, it was hoped that a propellant burning rate could be determined for each closed-bomb trial using the P-t curves and the changes in the grain dimensions of extinguished samples of plasma and conventionally ignited M30 and JA2 [4]. However, the fact that the P-t curves of the plasma-ignited grains show an initial rapid pressurization made the analysis more complicated.

With the interrupted burning tests, the approach used was to consider identical propellants, fired under similar plasma power conditions, for which the

pressure-time histories were comparable, with the exception that they were extinguished at two different pressures. The recovered grains were measured to determine the extent of regression between the two blowout pressures. Typically, during the plasma pulse, the pressure would rise to about 30 MPa; therefore, comparison of the calculated regression (using conventional burn-rate parameters over the experimental pressure interval) with the measured propellant regression between two pressures beyond 30 MPa yielded the extent of the intrinsic post-plasma burn-rate augmentation.

For both M30 and JA2, no evidence was found of increased burning rate after the plasma event, i.e., the measured grain regression is consistent with those predicted using conventional burning rates. For both M30 and JA2, grain regression at 35 MPa was consistently slightly greater than predicted based on conventional burning rates, suggesting that there is burning-rate augmentation during the plasma event. Thus, it appears that the post-plasma burn rates are not intrinsic [5].

Burning occurred to a greater extent in the samples ignited with black powder than with the plasma, because plasma-ignited samples had a significant fraction of the total pressure caused by the plasma injection. Thus, conventionally ignited samples are exposed to a longer burn time. This is reflected by the extent of grain regression. Average perf diameters of conventionally ignited samples of M30 and JA2 were 1.15 mm and 1.07 mm, respectively, while for plasma-ignited samples, the perf diameters were 1.07 mm and 0.98 mm, respectively. Another indication that burning had progressed to a greater extent in the conventional samples was apparent with the color change of the white virgin M30 grains to a golden-brown color in the samples extinguished after conventional ignition (Figure 3a). Most recovered grains from the plasma ignition were blackened (Figure 3b). However, on surfaces where the propellants were shielded from the direct blast of the plasma, the M30 grains from plasma-ignited samples (100 MPa) appear as a lighter cream color than golden-colored conventionally ignited samples (75 MPa) even though grain regression was comparable. This suggests a difference in chemical mechanism between the conventional and plasma ignition.

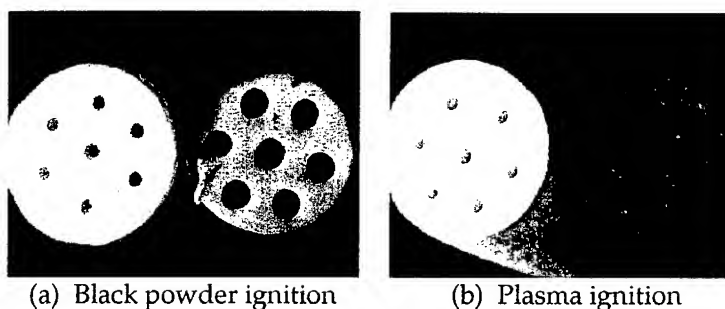


Figure 3. Photograph of M30 propellant samples extinguished at 75 MPa. A virgin (white) grain is shown with each.

3.2 FTIR Analysis

The IR spectra of virgin and extinguished propellant samples are shown in Figures 4a and 4b (JA2 and M30, respectively). Dominant bands in the spectra of both propellants at 1650 cm^{-1} and 1285 cm^{-1} are due to the nitrate-ester ($\text{O}-\text{NO}_2$) absorption. The JA2 microreflectance spectra show a slight shoulder at 1734 cm^{-1} due to aldehyde formation. Samples that visually appeared altered, as though reaction had occurred, exhibited the aldehyde band more so than surfaces that were not noticeably changed. Extinguished samples from either plasma or conventional ignition, microtomed to a depth of about $50\text{ }\mu\text{m}$, yielded spectra virtually identical to the virgin material (not shown). Thus, decomposition appears to be limited to surfaces less than this depth.

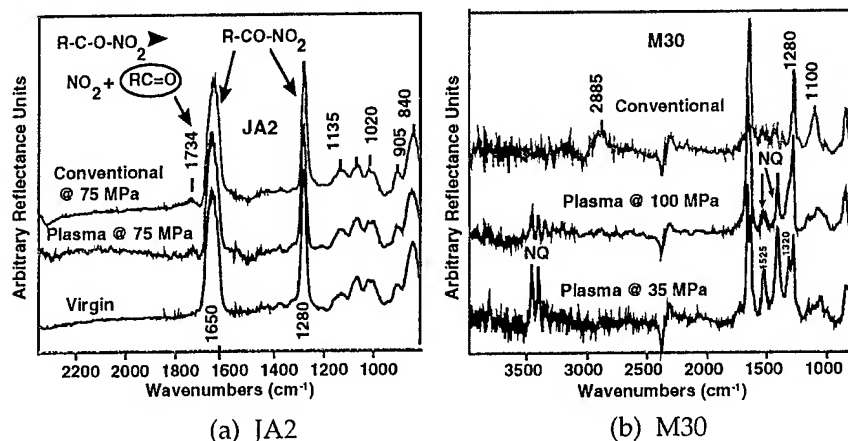


Figure 4. Microreflectance spectra of virgin, plasma- and conventionally ignited propellants.

In addition to the nitrate-ester bands, the spectrum of M30 has bands from nitroguanidine (NQ) as well. Bands due to NQ diminish after either plasma or conventional ignition, although the effect is significantly greater with conventional ignition, probably due to the greater extent of burning. The microreflectance spectra of the perforated regions of M30 grains were also obtained (not shown) for plasma-ignited samples extinguished at 35 and 100 MPa. The results are consistent with those from the outer surfaces of the samples, i.e., that at increased pressures (i.e., increased extent of burning), the NQ level is diminished. No difference in the NQ level within the perforated region was detected between the plasma and conventionally ignited samples. Also, in M30, a band at 1100 cm^{-1} (Figure 4b) from an unknown source becomes more distinct in the spectra of the extinguished grains, and is more prominent in spectra from conventionally ignited samples. This molecular species may also contain the aliphatic ($-\text{CH}_2$) and ($-\text{CH}_3$) groups, as evidenced by bands at 2885 cm^{-1} and 2975 cm^{-1} . Analysis by liquid chromatography-mass spectroscopy (LC-MS) could be helpful to isolate and identify the product.

3.3 SEM

Extinguished surfaces have been studied previously [6], and it has been noted that many combustion features are preserved on surfaces that undergo this manner of rapid pressure reduction. The flame is rapidly blown away from the surface, which quickly solidifies, leaving most of the burning surface features intact.

SEM micrographs revealed several interesting features. The exterior lateral surfaces of the extinguished grains from both the ignition sources appeared very similar except for one feature. In plasma ignition the grains showed evidence of burning caused by hot particles being sprayed onto the grain surface, as shown in Figure 5.

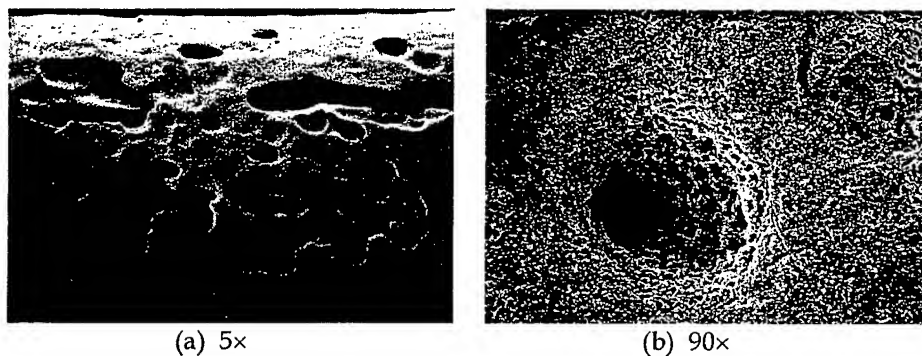
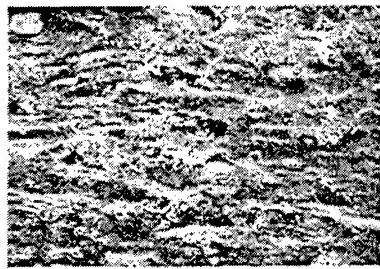


Figure 5. Micrographs of extinguished grain exterior showing evidence of hot particle spray from the plasma igniter (100 MPa).

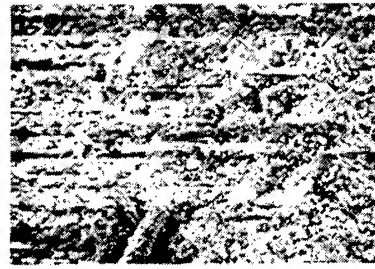
These features seemed to have further developed as the extinguishing pressures were increased. At higher extinguishing pressures, the grains burn for a longer period of time, so the progression of the burning can be followed. This indicates that perhaps the hot particles were projected through the surface and burning took place under the surface along the line of travel.

This phenomenon would tend to augment the surface area available and would give the appearance of a higher burning rate during the early portion of the propellant combustion. However, since the augmentation is caused by increased surface area, the apparent burning rate will be lowered in later stages of burning, as the resulting surface area is reduced due to the intersection (burn through and crossing) of burning surfaces.

Another interesting feature was noted on the burning surfaces within the perforations. Most burning surfaces of extinguished propellant appear as shown in Figure 6a, which depicts the conventionally ignited extinguished surface. Note that the surface is smooth, indicating melting. However, in Figure 6b there seems to be no indication of melting. There also appears to be a series of grooves



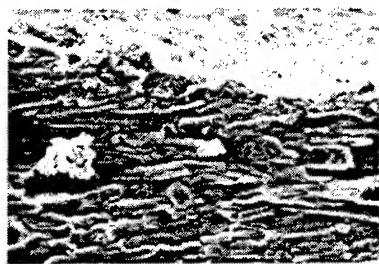
(a) Conventional Ignition
(70 MPa, 850×)



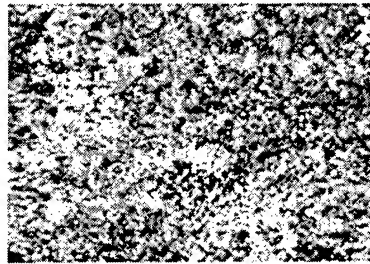
(b) Plasma Ignition
(35 MPa, 850×)

Figure 6. Micrographs of the burning surface of the extinguished grain perforation showing different features between ignition methods.

that conform to the size of NQ particles, which appear at the same magnification in Figure 7a. Stereoscopic pictures that were taken and analyzed confirmed that the features being observed were grooves. This indicates a different combustion process is in effect for the plasma-ignited samples that cause the NQ crystals to vacate the matrix. Note that in Figure 7b the grooves are much less prominent at higher pressure (100 MPa) and the surface appears more like the conventional. This is consistent with the burning mechanisms returning to similar processes at higher pressures. One other observation on the morphology of the M30 propellant is worth noting. While most propellants have measurable melt layers that range from 2–30 μm in thickness, the melt layer for M30 is so thin that it was not able to be measured. The evidence for a melt layer is shown in Figure 6a, as noted there. However, when a cross section of that surface is made and the thickness measurement is attempted, no thickness can be determined because it is so thin. The propellant goes from the outer surface to a structure identical to the unburned structure regardless of the ignition source without any measurable transition depth ($<0.2 \mu\text{m}$).



(a) Virgin (850×)



(b) Plasma Ignition (15 Kpsi, 850×)

Figure 7. Micrographs of the burning surface of the extinguished grains showing different features between ignition methods perforation.

4. Conclusions

The goals of this work were twofold: (1) to assess whether the plasma augmentation of propellant burning rate with plasma ignition is real or related to increased mass generation due to increased surface area (e.g., propellant fracture [3]) and (2) to study the mechanisms of plasma-propellant interaction using analytical techniques of SEM and FTIR spectroscopy. For either M30 and JA2, no evidence was found of increased burning rate after the plasma event, i.e., the measured grain regression of the extinguished propellants is consistent with that predicted using conventional burning rates. For both M30 and JA2, grain regression at 35 MPa was consistently slightly greater than predicted based on conventional burning rates, suggesting that there is burning rate augmentation during the plasma event. Work is in progress to assess the vivacity curves for the conventional P-t data obtained in this work, and to simulate the effects of fracture on these curves [3, 5].

The goal of understanding the interaction of plasma with M30 and JA2 propellants, as well as early combustion events with conventional ignition, is only beginning to be realized by this effort, although some important lessons on how to proceed in the future have been learned. For propellants such as JA2 and M30 which contain high levels of plasticizer (e.g., diethylene glycol dinitrate [DEGDN], nitroglycerin [NG]) that migrate, analysis is complicated by the fact that, even if the reaction at the surface is appreciable, the bulk of the propellant remains unreacted and diffusion of the remaining plasticizers after extinguishment is rapid which masks the effects of decomposition. It is noted that in previous studies of these propellants, no significant change in the Akardite II or plasticizer level was detected for grains extinguished from either plasma or conventional ignition [7]. For recent extinguished closed-bomb studies of JA2 and M30 with mylar capillaries, propellant surfaces were sampled immediately for analysis of decomposition products by the more-sensitive LC-MS (work in progress).

Extinguished M30 grains (conventionally ignited, 75-MPa blowout) burned to a greater extent than those ignited with the plasma. Moreover, M30 grains appear almost white in the case of plasma ignition (except for areas that received the direct blast of the plasma, which were blackened), and a golden-brown color for samples that were conventionally ignited, even in samples for which grain regression was comparable. Extinguished grains of M30 show that the NQ diminishes in plasma-ignited samples but is virtually eliminated with conventional ignition. Moreover, an unidentified decomposition product, apparently with aliphatic C-H, is evident in extinguished grains of conventionally ignited M30, which is less apparent when plasma ignition is used.

Extinguished JA2 grains (75-MPa blowout), ignited with either plasma or black powder, both show evidence of aldehyde formation due to denitration of the nitrate esters. Thus, under these conditions, denitration appears to be the result of burning, as opposed to direct interaction of the plasma with the propellant. It is noted that in other experiments, exposure to only plasma radiation resulted in denitration and plasticizer depletion due to subsurface reaction of the propellant [8].

The SEM micrographs revealed that differences exist between the morphology of the burning surfaces ignited by conventional means and by the plasma. The plasma sprays hot particles into the surface exposed to the plasma blast, which results in unprogrammed surface combustion. In addition, the surface features within the perforations indicated that different combustion mechanisms were active there. In the conventional ignition, a melt layer was indicated (smooth surface), whereas in the plasma ignition it appears that in some regions NQ crystals near the surface appeared to be absent and there was no indication of a smooth melt surface. The main conclusion from the morphology investigation was that the burning process at lower pressures appears to have differences. However, as the pressures increase the perforation surfaces are observed to become more and more alike.

The extinguished closed bomb has provided a system for assessing propellant burning rates during and after exposure to plasmas, and for studying plasma-propellant interactions under well-controlled conditions of plasma power, energy, and proximity to the propellant, and propellant temperature and blowout pressure.

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